Non-uniqueness of the viscoelastic properties determined by conical nanoindentation. Case of polypropylene

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ABSTRACT

The complexity of the analysis of nanoindentation data when the materials exhibit a viscous behavior asks the fundamental question of the uniqueness of the properties determined by an analysis of the temporal evolution of the indentation load $P(t)$ and the indentation displacement $h(t)$. This problem of non-uniqueness of the properties obtained is often provoked in practice by a high sensitivity to measurement errors $P(t) - h(t)$. This difficulty is well known in the inverse analysis of the elasto-plastic indentation curve [1], it is on the other hand, little approached in the presence of viscous phenomena (viscoelastic and / or viscoplastic, etc.) as may be the case for polymers.

This study focuses on the possibilities of extracting viscoelastic properties (VE) of a polypropylene (PP) by conical nanoindentation. The objective is to develop a method for analyzing the $P(t) - h(t)$ data allowing to determine intrinsic and reliable VE properties (unique and stable). For this, a 2D-axisymmetric finite element model (EF) of the nanoindentation test integrating VE behavior has been developed [2]. This model has four parameters: Young's modulus E, Poisson's ratio ν, anelastic modulus c_1, and viscosity coefficient η. Nanoindentation load-unload tests to a depth of approximately 550 nm were performed on a PP sample for different rates of penetration of the indenter (50, 500, 1000 and 5000 nm/min).

It is found that it is possible to recalibrate perfectly the numerical model for each of the indentation rates, but that the values of the estimated VE parameters are very different from one rate to another. It is also found that very different combinations of parameter values can lead to a quasi-similar $P(t) - h(t)$ evolution. This result demonstrates the non-uniqueness of the values of the four VE parameters determined using a single load-unload test.

In order to quantify the richness of the information contained in the temporal data, an identifiability analysis was conducted. Based on the calculation of an identifiability index [3],

it shows that only two parameters can be identified from a load-unload test and that the major problem is related to the high sensitivity of the solution of the inverse problem to the uncertainties on the $P(t) - h(t)$ data.

To identify more than two parameters, it is essential to enrich the experimental information. An identifiability analysis using simultaneously data from several tests is currently underway. The interest of taking into account a hold step will be quantified by the same method. This approach based on the parametric identifiability should make it possible to design a test sufficiently rich to determine intrinsic and reliable values of the four parameters VE, and in the medium term to extract viscoelastic and viscoplastic properties of massive materials or thin films.

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